



PATENT SPECIFICATION

NO DRAWINGS

1,006,706

Date of Application and filing Complete Specification: Oct. 9, 1961.

No. 36209/61.

Application made in United States of America (No. 65302) on Oct. 27, 1962.

Complete Specification Published: Oct. 6, 1965.

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Index at acceptance:—A5 B(31, 32, 33, 34); C5 D(6B2, 6B6, 6B10B, 6B12B1, 6B12E, 6B12F1, 6C5, 6C9)

Int. Cl.:—A 61 k 7/00 // C 11 d

COMPLETE SPECIFICATION

Cosmetic Preparations

- We, FMC CORPORATION, of San José, California, United States of America, a corporation organized and existing under the laws of the State of Delaware, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—
- This invention relates to cosmetic preparations or compositions.
- According to the present invention, there is provided a cosmetic composition containing cellulose crystallite aggregates as hereinafter defined having an average level-off D.P. in the range of 15 to 375 anhydro-glucose units and at least one active cosmetic ingredient compatible with the aggregates and present in the composition in an available form and in an amount sufficient so as to impart its characteristic cosmetic effect or property when the composition is applied topically.
- The term "cosmetic" as used herein is intended to embrace all types of products which are to be applied in any manner directly to the person for the purpose of cleansing or embellishment, including altering the appearance. Detergent compositions, toilet soap, bath additives or bubble baths, face masks and shaving soaps and creams, for example, are intended to be included in this definition as well as, for example, deodorants, depilatories, and suntan and sunscreen preparations.
- The term "cellulose crystallite aggregates" as used herein means a product obtained by the acid hydrolysis of cellulose, there being formed an acid-soluble portion and an acid-insoluble portion. The latter comprises a crystalline residue or remainder; it is washed and recovered, being referred to as "cellulose crystallite aggregates", or as level-off D.P. (degree of polymerization) cellulose. The preferred aggregates for use in this invention, their preparation by the acid hydrolysis of cellulose, their properties and the mechanical disintegration of such aggregates are described in our Patent No. 874,945 and in the Complete Specifications of our co-pending Applications Nos. 29671/60 (Serial No. 970,111) and 29672/60 (Serial No. 970,112).
- The level-off D.P. value reflects a destruction of the original fibrous structure of the cellulosic source material. The term "level-off D.P." has reference to the average level-off degree of polymerization of the cellulose products measured, for example, in accordance with the paper by O. A. Battista entitled "Hydrolysis and Crystallization of Cellulose", Vol. 42, INDUSTRIAL AND ENGINEERING CHEMISTRY, pages 502—7 (1950).
- The crystallite aggregates are available in a high state of purity being one of the purest forms of cellulose, and are not likely to cause undesirable dermatological reactions. They have a smooth texture, being obtainable in the form of impalpable powders, and are substantially opaque. They are further characterized by having a variable particle size ranging from below 1 micron to 250 to 300 microns. They are obtainable in batches of varying particle size, or of very uniform particle size; in other words, the particle size is controllable.
- The cosmetic preparations, aside from the presence of the crystallite aggregates, may be generally those in current use, for which the aggregates provide many useful advantages, depending on the cosmetic preparation.
- By virtue of their unique properties, the crystallite aggregates are capable of improving a wide range of cosmetic products. In particular, the aggregates possess the highly useful property of forming stable homogeneous colloidal gels and dispersions; when suitably mixed with cosmetic ingredients, the use of the aggregates makes possible the production of stable gels and dispersions without requiring the presence of an emulsifying agent, and these gels and dispersions, which may be generically classed as suspensions, may themselves comprise, or are readily adapted to produce,

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cosmetic creams, lotions, emulsions, dispersions, pastes, cakes, sticks and the like.

The aggregates have a substantial affinity or sorptive power for oily, fatty and waxy materials, and also for water and other aqueous fluids, and for these reasons are of value in binding and stabilizing these and other ingredients (for example, water-insoluble preparations such as magnesium carbonate, for instance) of a preparation. In many cases, free-flowing apparently dry granular mixtures may be formed by mixing the aggregates with an oleaginous or aqueous material; the resulting mixtures may be useful *per se*, or may simplify the formulation process as by serving to introduce to the formulation such agents as flavours, perfumes, and the like, which are usually employed in low concentrations.

The aggregates are compressible, and this property is imparted to mixtures containing them with the result that cosmetic forms like cakes and sticks are conveniently prepared. Coatings deposited from aggregates-containing cosmetics are easily washable from the skin or other surface by water.

The source material for the cellulose crystallite aggregates may be any of the natural cellulose materials, such as natural fibres, for example, ramie, cotton, purified cotton, purified wood pulps such as bleached sulphite pulp or bleached sulphate pulp, or regenerated forms of cellulose, for example, rayon or the product sold under the registered trade mark "Cellophane", and in all instances has an actual D.P. greater than its average level-off D.P. The aggregates have an average level-off D.P. value in the range of from about 15 to about 375 and the specific value will be dependent upon the source material. Aggregates having an average level-off D.P. in the range of 15 to 60, for example, are produced from regenerated forms of cellulose. Aggregates having an average level-off D.P. in the range of 60 to 125 may be obtained from alkali swollen natural forms of cellulose such as cotton linters and purified wood pulps. Sulphite pulp as a source material will produce cellulose crystallite aggregates having an average level-off D.P. in the range of 200 to 300. Although the cellulose chains in the crystallites are uniform in length by comparison with the source material, some variation occurs, and, for this reason, the D.P. is referred to as an average level-off D.P. value.

In accordance with recognized principles of polymer chemistry, in speaking of degree of polymerization (D.P.), reference is made to the pure polymer. It is to be understood that the stated level-off D.P. values of the cellulose has reference to substantially pure cellulose such as cellulose crystallites derived from highly purified cellulose sources including regenerated forms of cellulose. Where a crude or raw wood pulp is subjected to a hydrolysis treatment for the production of the level-off

D.P. cellulose crystallites, appreciable amounts of lignin and other non-cellulose materials will be present in the residue and measurements of average level-off D.P. of such residue may exhibit apparent values as high as 500. Such high values reflect the presence of lignin and other non-cellulose material and are not true values of the level-off D.P. of the cellulose *per se*. However, there is present in the mass some cellulose which has been reduced to the average level-off D.P.

Associated with the level-off D.P. properties of the crystallite aggregates is the known fact that their chemical purity is very high and these aggregates constitute perhaps the purest form of cellulose. For example, the ash content ranges from about 10 to 600 ppm. whereas conventional fibrous cellulose will have an ash content of from 1000 to 4000 ppm.

It is well known that the various forms of cellulose, such as cotton and wood pulps, contain non-cellulosic components including plant steroids which steroids are believed to be chemically bound to the cellulose and thus are in a physiologically inert state. For example, purified wood pulps commonly termed "high alpha pulps" contain from about 1000 ppm. to about 3000 ppm ether extractable components including plant steroids which cannot be assimilated by the human system. Acid hydrolysis in disrupting the fine structure of the cellulose may make the plant steroids more available for physiological assimilation. Cellulose crystallite aggregates, for example, formed from a refined wood pulp (93% alpha cellulose, 1000 ppm. ether extractable components) will contain about 800 ppm. of the ether extractable components. For certain preferred purposes of the present invention, the ether extractable component content (including the steroids) may be from about 50 ppm. up to about 500 ppm., preferably not exceeding about 200 ppm. Cellulose crystallite aggregates formed from high alpha cellulose and, for example, containing 800 ppm. of ether extractable components may be subjected to hot isopropanol extractions to reduce the extractable component content to less than 200 ppm. Special pulps of a dissolving type are commercially available containing less than 500 ppm. of ether extractable components including plant steroids and obviously the cellulose crystallite aggregates formed from such cellulose sources will have the desired low ether extractable component content.

In the production of the cellulose crystallite aggregates, they are washed with water after hydrolysis of the source material and the particle size of the individual aggregates will vary from about one micron to about 300 microns as determined by visual microscopic examination. These aggregates may be mechanically disintegrated to form small disintegrated aggregates on products having a particle size less than one micron to about 300 microns

and the particle size and the particle size distribution may be varied depending upon the specific disintegrating method and the period during which the aggregates are subjected to disintegration.

5 The preferred disintegration method is to attrite the aggregates, that is, subject the aggregates to a rubbing or shearing action, by means of a high speed cutting action in the presence of an aqueous medium. It is preferred that the water content of the mixture undergoing attrition should be at least about 15% to 25% by weight. The aggregates content of the mixture to be attrited should be at least 3% by weight and, desirably, is higher as the efficiency of the cutting action increases with the aggregates content. For example, attrition at a 3% concentration so as to provide at least 1% of a particle size less than one micron will produce a milky dispersion. The relative viscosity or thickness of the dispersion varies with the relative proportion of particles of a size less than one micron. Prolonging attrition will increase the amount of fine particles and, accordingly, increase the viscosity to some extent. The stability of the dispersions increases with an increase in the pH of the medium. At pH's between substantially neutral and 11, the dispersions remain stable indefinitely.

10 Either before or after mechanical disintegration, the aggregates may be dried. For some purposes, it is preferable to de-water rather than dry the material because the never-dried cellulose crystallite aggregates form dispersions more readily and the dispersions have a smoother texture when prepared from a never-dried material. The aggregates may be dried and recovered in particulate form, for example, by freeze drying, spray drying, drum drying, vacuum drying and drying by solvent displacement.

15 A particularly unique and distinguishing characteristic of the disintegrated aggregates is that a stable dispersion of the aggregates in an aqueous medium, where the aggregates constitute about 1% by weight of the dispersion and at least 1% of the weight of the dispersed aggregates have a particle size of up to one micron, when applied to glass and the water allowed to evaporate forms an extremely adherent film on the glass.

20 For the purposes of the invention, a dispersion may be defined as having about 1 to about 8% by weight of the aggregate dispersed in the aqueous or other liquid, the latter constituting the continuous phase of the mixture. The dispersion has the physical form or appearance of a liquid, and is flowable like a liquid. A gel may be defined as having about 3 to about 35% by weight of aggregates dispersed in the aqueous or other liquid, and in this case the aggregates constitute the continuous phase of the mixture. The gel has the physical form of a jelly, paste, plastic mass or

the like. Both dispersions and gels are included by the term suspension.

In general, and as has been indicated, the aggregates are preferably attrited before being used to make up a cosmetic composition. In some instances, as in the manufacture of face powders, the aggregates need not necessarily be attrited, particularly if they have been dried as by spray drying, although in general attrited aggregates are preferred. It will be apparent that in the case of water-containing cosmetic compositions, the aggregates may be initially in the form of wet gels which may serve to provide the water content of the composition as well as to aid in obtaining a well-mixed product. For dry compositions, the aggregates may be employed in dry form, and the same is true where they are to be used in non-aqueous oleaginous preparations. For the latter, the aggregates are advantageously premixed with the oily, fatty, and/or waxy material to form an apparently dry, free flowing particulate solid or "crumb," thus facilitating the formulation of the product.

A variety of cosmetic preparations may be benefited by the addition of the aggregates, although as they are water- and oil-insoluble, their use is limited to preparations at least partly composed of solid materials.

25 Considering the application of the aggregates to specific cosmetic products, they are useful in creams, which essentially comprise an oleaginous base, either as an addition thereto or to replace, at least in part, oily, fatty, and/or waxy ingredients of the cream. For example, the aggregates may partly replace the almond oil, mineral oil, lanolin, beeswax, paraffin wax, oleic acid, or spermaceti, and the like which are conventionally used in creams, whether of the cleansing, emollient, or finishing types, and including cold cream, quick-liquefying cream, liquid cleanser cream, night cream, massage cream, vanishing cream, foundation cream, and various special creams. An advantage of replacing at least part of such materials is that the soiling tendency of the creams is reduced, that is to say, the creams after being spread over the skin by the user are less apt to pick up or attract soil or foreign particles; similarly, the deposited creams transfer off the user's skin less readily, as by contact with clothing, bed sheets, and the like. This last advantage is of particular importance in other creams such as deodorants, including deodorizer and anti-perspirant creams, which are used under the arms and on the palms and soles and which are quite apt to come in contact with clothing and to soil it to such an extent that the garments are frequently ruined. The glycerol content of finishing creams such as vanishing or foundation creams may be partly substituted by the aggregates to provide a resultant cream of increased opacity or whiteness, which many consider to be of better appearance than creams which tend to have

a translucent or pearly luster. However, creams of the latter type may be made by holding down the aggregates content. The lanolin, petrolatum, ceresin, beeswax, cocoa butter, and/or stearic acid contents of emollient and vanishing creams, and also of cream and paste rouges, may be partly supplanted to reduce their soiling tendency, and more particularly to reduce their oily or greasy feel while yet retaining their power of free motion over the skin. Because they are lubricitous, and confer lubricity on a cream preparation without increasing its oleaginous character, the aggregates are suitable for addition to creams to be applied to the oily skin. Straightforward addition of the aggregates to many creams is of value where a stiffening effect is sought.

Special creams such as protective creams may usefully incorporate the aggregates, as by addition, and the cream may be of any suitable type, such as a mixture of fats and oils, a jelly containing a physical barrier, an emulsion, or a soap base. These creams are of particular value for protecting the hands from injury, or from soiling, in the carrying out of many processes and procedures in industrial plants. Those creams containing physical barriers are particularly adapted to receive the aggregates, which, being available in varying particle sizes, may form protective barriers on the skin of varying degrees of fineness and smoothness.

In cleansing lotions, which usually comprise more or less permanent suspensions of mucilaginous substances, conventional abrasives such as magnesium carbonate may be replaced in whole or part by the aggregates to reduce or eliminate the problem of settling. The magnesium carbonate, being insoluble and heavier than water, tends to settle out of the lotion on standing, an occurrence that detracts from the appearance of the preparation and which requires the user to shake the lotion well before each use. The use of the aggregates may not only provide an abrasive, but also, owing to their stable gel- and dispersion-forming properties, may lead to an indefinitely stable homogeneous lotion and may thus make unnecessary the need for conventional emulsifying agents like borax. It will be appreciated that the aggregates may at least partly replace the use of other metal salts and oxides which have a tendency to settle out of a lotion or suspension. Thickening of lotions, and also creams, may be readily achieved by simple addition of the aggregates and without relying on special agents like agar, pectin, and the like.

Make-up powders for the face may benefit from the presence of the aggregates. These powders usually comprise an opacifying agent (clay, titania, magnesia, zinc oxide, etc.), a slip material (talc, metal stearates, etc.), and adherent material (stearates, clay, etc.) and an absorbent (chalk, calcium carbonate, kaolin,

etc.). The aggregates are capable of replacing, in whole or in part, metal salts and oxides like chalk, kaolin, magnesium carbonate, talc, titania, magnesium stearate, zinc oxide, zinc stearate and the like. An advantage of such substitutions or replacement is the reduced incidence of undesirable dermatological reactions. For example, in the case of magnesium carbonate, a widely used ingredient, it is known that some persons are sensitive to the presence of trace amounts of elements like selenium, arsenic, or mercury which tend to be present in the magnesium carbonate owing to the difficulty of purifying it. The gravity of the problem is underlined by the fact that amounts of selenium as low as one part per million may be detrimental. Other conventional ingredients, if not sufficiently fine, may give rise to mechanical irritation. In the case of ingredients like talc, by omitting it there is eliminated its characteristic earthy odour, which otherwise must be masked. Of further interest is the effectiveness of the aggregates for sorbing oil, this property being useful in face powders for combating oily skin, especially on the nose. By sorption is meant the capacity of the aggregates to absorb and/or adsorb oily, fatty, greasy, waxy and aqueous materials. Starch may be replaced in loose powders, and the resistance to caking of the powder may thus be improved in view of the fact that the aggregates per se are much more resistant to caking than starch. Starch becomes doughy even from perspiration.

In addition to the foregoing capabilities, the aggregates are of benefit to loose face powder compositions by virtue of their excellent adherence to surfaces, including the skin, and on this account are useful in place of the adherent agents noted; by the degree to which their particle size may be varied; and by the extent to which their fluffiness or bulk density may be changed. The particle size for use in powders may be as low as 1 or 2 microns; although more usually it may be 30 microns which corresponds to a powder capable of passing through a 400 mesh screen. They are also available in particle sizes as high as 250 to 300 microns, and, furthermore, in uniformly-sized fractions or batches. The apparent or bulk density may be as low as 7 or 8 lbs. per cu. ft., which represents a very fluffy material, and as high as 34 lbs. per cu. ft. By comparison, conventional corn starch has a bulk density of 36.8 lbs. per cu. ft. By virtue of their fluffiness, and depending on the amount of aggregates incorporated in a preparation, the conventional step of aerating face powders before packaging may in some cases be omitted. In the lower particle sizes, say up to 30 or 50 microns, the aggregates comprise an impalpable, easily distributable or rubbable powder well suited to the compounding of even the most delicate cosmetic products. In addition to having the foregoing properties, the

- aggregates may be given a variable degree of opacity by suitable selection of particle size, as by blending fractions of different sizes; thus the covering power of a face powder may be varied. It will be apparent that by themselves the aggregates possess most if not all of the requisite properties of a face powder. By incorporating conventional amounts of a desired colour and a desired perfume, a complete, esthetically suitable face powder may be made which will inherently have a matte effect, and which may be used *per se* or serve as a base, with or without the colour and perfume, and to which only minor additions need be made to obtain desired finished powders to suit different types of skin.
- In compact powders, including face powder and rouge, which are pressed after the addition of a binder such as, for example, gum arabic, gum tragacanth, glycerine or sorbitol, the use of the aggregates may eliminate the need for the binder as the aggregates are inherently compressible in the dry or wet state to any desired degree. As with loose powders, the aggregates may serve as a base for making various types of compact powders and in this form may comprise the major portion of the finished product.
- The aggregates are also useful in lipstick compositions and may replace one or more oleaginous ingredients in whole or part, such as the fats and waxes, with the advantage of decreased greasiness without loss of lubricity, and improved consistency retention. Fats and waxes commonly used in lipstick compositions include beeswax, carnauba wax, ceresin, lanolin, lard, mineral oil and petrolatum. Conventionally used lipstick flavors, usually comprising a volatile, water-immiscible organic ester, may be better retained in the lipstick owing to the good sorptive capacity of the aggregates for such compounds. The aggregates form free-flowing, apparently dry mixes with flavors, and thus may facilitate lipstick manufacture by serving as a carrier for introducing these volatile compounds to the production batch and losses of the flavors may be reduced.
- In mascara compositions such as mascara cake, the aggregates may supplant conventional soaps such as, for example, triethanolamine stearate and triethanolamine oleate, and thereby render the preparation less irritating to the eyes; furthermore, the cake itself may be prepared more easily owing to the compressibility of the aggregates. The waxes frequently used in these preparations, and also in roll-on mascara and cream mascara, may be at least partly replaced, with advantages similar to those described in wax substitutions. On account of their fine particle size and good adherence, the aggregates are capable of imparting a cleaner effect to eyelashes, avoiding the thick, pasty or crumbly look resulting from the use of some conventional preparations or the startling effect imparted by some enamel-like preparations. Use of the aggregates permits a wider selection of colors to be employed and thus may avoid dependence on the conventional but somewhat dangerous use of silver nitrate with sodium thiosulphate. Suitable colours may include natural pigments, including carbon, ochers, siennas, umbers and ultramarine. In similar ways, the aggregates may be of value to other cosmetics for use around the eye, as in eye shadow sticks, eye liner pencils, and eyebrow pencils.
- The aggregates may replace certain components in depilatories, particularly earthy fillers like talc and lime, or whiteners like barium sulfate, magnesnia, and titania, with advantages similar to those described.
- Particularly in the case of deodorant compositions like deodorizers and anti-perspirants, which frequently simply comprise an active deodorant ingredient and a cream base, the aggregates by themselves may constitute the base. For example, a deodorizer may be prepared by mixing one or more active agents such as, for example, hexachlorophene, bithional, salicylic acid, zinc peroxide, zinc phenolsulphonate, boric acid, benzoic acid or hydroxyquinoline sulphate, with the aggregates in gel form, and the resulting mixture, having an appearance, feel, lubricity, spreading quality, and adherence similar to a cream preparation, may be used as the deodorizer, or in many cases as a basic mix requiring only minor additions to make a finished product. Such a mixture or product has the advantage of being lubricitous without greasiness. If used without modifications or additions, it would, after application, readily dry to form a fine-grained, adherent coating easily removable with water. The aggregates in this coating will not soil or otherwise affect a fabric or garment with which it may come into contact. Similarly, an anti-perspirant composition may be prepared by mixing one or more conventional active agents such as, for instance, alum, aluminum chloride, zinc chloride, aluminum acetate, zinc sulphate, zinc phenolsulphonate, tannic acid and tannin, with the aggregates in gel form. Both aqueous and non-aqueous aggregates gels are of value in the foregoing compositions.
- The crystallite aggregates may improve shaving creams, soaps and sticks of the lathering type by strengthening and stabilizing the lather. It appears that the tiny particles of aggregates strengthen the gas-enclosing walls of the lather and prevent them from readily breaking down. The aggregates may replace, at least in part, the conventional talc whitener used in some powdered shaving soaps and in styptic pencils and thus may avoid or reduce the characteristic earthy odour of the talc. In powdered soaps the aggregates may also function to prevent caking.
- In sunscreen compositions, the aggregates, being opaque, may substitute for conventional

opaque ultraviolet ray-scattering agents like talc and titania, with advantages already described. In these compositions, as well as in many others, including suntan products and leg paints, the aggregates provide lubricity without greasiness, and in addition, the adherence of the product to the skin is enhanced, and its water-removability improved. As these compositions are frequently in dispersion form, a further advantage resides in the stability which may be imparted to the dispersions by the presence of the aggregates. Of interest in this connection are simple 2-, 3-, or 4-component sunscreen compositions made by mixing a base like petrolatum or zinc oxide or lanolin with an aggregates-containing gel having, say, 3 to 25% by weight of aggregates and the balance water. A sunburn preventive may be added to help block out harmful radiation, including such agents as acetanilide, cholesterol, p-aminobenzoic and salicylic acid salts, quinine salts and tannins. These components may form compatible mixtures. Suntan make-up, whether in loose powder form for the face and other areas, or in cake form, may be benefited in the manner described for make-up powders.

In manicure compositions, water removable nail coating compositions may be prepared comprising simply the aggregates, a colouring agent, and water, and in which the aggregates, particularly of the lowest particle sizes, provide the necessary adherence to the nails. Such compositions are of value for application to the nails for a single occasion of short duration, after which the coatings are removable by simply washing the hands in water. Being safe, they may be used by children.

Facial masks may be improved in respect of their adherence and water-removability by the addition of the aggregates, which furthermore, may replace such less desirable inorganic components as bentonite, fuller's earth, kaolin and infusorial earth.

Bath additives or bubble baths, may be benefited by the aggregates in respect of the stability of the resulting bubbles. As noted, the aggregates have stabilization properties for bubbles and lathers. They may replace conventional stabilizers like methyl cellulose which tends to form viscous gummy solutions. In fragrant bath powders or dusting powders, the aggregates may substitute for conventional talc, and colloidal clay, as absorbents, and may replace titania as a whitener. They are further able to replace adherence-improvers like magnesium or zinc stearates. In other fragrance preparations, the aggregates may be mixed with a perfume to provide an apparently dry, solid, free-flowing, fragrance mixture, or sachet, suitable for sprinkling per se in linen closets, clothing drawers, and the like, or for use in sachet bags. The aggregates retain the perfume so that the fragrance is slowly given up over a period of time. Simi-

larly, other solid forms like sticks, compressed cakes and the like may be prepared.

The cellulose crystallite aggregates are also utilized advantageously in detergent compositions. These compositions comprise cellulose crystallite aggregates and an active detergent substance such as the usual soaps (alkali-metal salts of higher fatty acids) and the wide range of synthetic surface-active agents which have replaced soaps for various purposes. The detergent substance is present in the composition in an amount sufficient to impart its detergent characteristic to the composition. For example, in certain toilet soaps, the aggregates may be used in lieu of all or part of the coconut oil, which, although widely used as a lathering agent, is considered to be harsh and irritating to the skin. In this substitution, the aggregates, may be of 30 to 100 microns particle size, again impart lubricity without greasiness, and they improve the lather strength. They may also act as a whitener and as an abrasive in bar and powdered soap, and prevent the latter from caking.

Powdered dentifrice preparations may be rendered anti-caking by the aggregates, which also retain the flavor better.

Not the least of the characteristics of the aggregates is their removability from the skin by the application of water, regardless of the preparation in which they are used. While they are not soluble in water, they are dispersible in it and are readily washed off the skin.

Any preparation in which settling or sedimentation may be a problem may be aided by the addition of the aggregates, it having been found that their stable dispersion- and gel-forming characteristics are also applicable to more complex mixtures. Elimination of settling is particularly possible in preparations where gels of the aggregates may be used owing to the higher content of aggregates which gels may have.

As indicated, many of the cosmetic ingredients which the aggregates are capable of supplanting are toxic to some extent. Thus, magnesium oxide and magnesium carbonate exhibit a moderate local irritant effect, either acute or chronic, on the skin or mucous membranes; while calcium carbonate, paraffin wax, triethanolamine, oleic acid, castor oil and palm oil all exhibit a slight irritant effect. Caustic soda and caustic potash have a high irritant action. Almond oil, beeswax, lanolin, spermaceti, ceresin, carnauba wax, castor oil, starch, tragacanth gum, and palm oil have been recognized as having a slight allergenic effect. Talc, borax and caustic soda exert a moderate local toxic action when inhaled, while with calcium carbonate, triethanolamine and starch, the action is slight. When ingested, borax has a moderate toxic effect and calcium carbonate and triethanolamine have a slight effect. Caustic soda and caustic potash have a high toxic action when ingested.

On the other hand, the crystallite aggregates are safe enough to be eaten and have no known toxic local effects. They have a bland taste and odour, a white colour, and physically resemble starch.

With the exceptions noted, the methods of formulating the cosmetic compositions may, in general, be those employed for conventional cosmetics or other similar commercial products. A convenient and effective method, in some cases, is to start with the aggregates in gel or dispersion form and to blend the other ingredients therein; if a dry powder is desired, the resulting blend may be subjected to spray drying.

The invention may be illustrated by the following examples.

The cellulose crystallite aggregates used in the succeeding examples were prepared by hydrolyzing Ketchikan sulphite wood pulp with a 0.5% by weight aqueous solution of HCl for 1 hour at 250°F., there being obtained a material having an average level-off D.P. of 220 and a purity above 96%. This material was dried in a vacuum oven at 158°F. to a moisture content of about 4 to 5% by weight. It was then screened, and the fraction passing through a 325-mesh sieve (44 microns and less) was taken.

EXAMPLE 1

A conventional cleansing cream was prepared, sample C and also an identical cream, D, but containing crystallite aggregates, the compositions of which were as follows:

	C	D
Beeswax	8.4 g.	8.4 g.
Mineral oil	25.0	25.0
Borax	0.4	0.4
Water	16.2	16.2
Crystallite aggregates	—	5.0

Both creams had substantially the same lustrous pearly white appearance, although upon standing, water separated from cream C. A small amount of liquid in the form of a milky dispersion also separated from cream D, the amount of which was considerably smaller than in the case of C. Both creams were easily movable over the skin. After removal from the skin by wiping with absorbent paper, cream D appeared to leave the skin with a smoother feel.

EXAMPLE 2

A hand lotion was made up comprising:

Crystallite aggregates	15 g
Glycerine	73
Distilled water	10
Oil of peppermint	2
Sodium lauryl sulphate	trace

The lotion was viscous but readily pourable and had a lustrous pearly white appearance. It rubbed smoothly over the hand, giving the impression of lubricity without greasiness; on drying the applied coating, no visible trace of the aggregates was apparent.

EXAMPLE 3

An anti-perspirant composition was made up containing:

Stearic acid	16.0 g
Crystallite aggregates	5.0
Sodium lauryl sulfate	1.5
Propylene glycol	5.0
Water	49.0
Urea	5.0
Aluminum sulphate	18.0

In formulating, two mixtures were prepared, one containing the aggregates and the stearic acid, and the other comprising the balance of the ingredients. Each mixture was heated to 70°C. and then mixed. The resulting product was in the form of a soft white odourless paste that was easily spreadable over the skin and had a non-greasy feel. It could be wiped off the skin readily with absorbent paper. After application to the skin and drying, the coating had the form of a white adherent non-greasy layer that was easily removed by wiping.

EXAMPLE 4

A shaving cream was prepared from the following:

Crystallite aggregates	50 g.
Triethanolamine	10
Oleic acid	20
Sodium lauryl sulphate	10 mg.
Water	q.s.

The ingredients were all thoroughly mixed together in a Waring Blendor, producing a cream which was placed in a bottle and capped. It had a light, wet appearance resembling conventional lather type shaving cream and had a pleasant odour. The preparation had a slippery, creamy feel on the skin without being greasy and was readily washed away with water. It was suitable for use as a brushless shaving cream.

EXAMPLE 5

A conventional calamine-containing cream-type sunscreen composition was made, identi-

fied below as E, and at the same time another preparation, F, was prepared in which crystallite aggregates were substituted for the calamine.

	E	F
Calamine	7.5 g.	—
Petrolatum	13.7	18.7 g.
5 Lanolin	6.2	6.2
Water	17.5	17.5
Crystallite aggregates	—	7.5

Upon being spread over smooth paper, cream F produced a coating that was noticeably less greasy than E. Also, cream F was white in colour and less apt to leave a visible stain on clothing, whereas E was pink owing to the

presence of the calamine.

EXAMPLE 6

Two powdered dentifrices, G and H, were made up having the following compositions:

	G	H
Sodium metaphosphate	38.9 g.	38.9 g.
Calcium carbonate	10.0	—
15 Sodium lauryl sulphate	0.5	0.5
Flavour	1.0	1.0
Saccharin	0.1	0.1
Crystallite aggregates	—	10.0

Sample G containing calcium carbonate as an abrasive was a conventional material, while in sample H the crystallite aggregates were used in lieu of the calcium carbonate. Both samples were thoroughly blended and then placed in stoppered bottles. After standing for several days, both were repeatedly smelt, and it was apparent that the aggregates-containing sample exhibited a stronger odour of the flavour (peppermint), although as made up both samples originally contained the same amount of flavour. When rubbed with the finger, both preparations had an abrasive feel, sample H being less abrasive than G. In appearance, sample H had a softer look and appeared to have a somewhat fluffy character not exhibited by G, which was a white conventional-looking powder.

As indicated, the aggregates are suitable for use in cosmetic compositions which contain an ingredient in the solid state; that is to say, a solid ingredient other than the aggregates. Where their presence is not harmful, they may be of benefit to non-solid-containing liquid preparations such as lotions, emulsions, solutions, liquid creams, or other liquid mixtures.

While the aggregates are normally in a solid state, whether dry or suspended in a liquid such as water, they are capable of existing in a form where some question may arise as to whether or not they are in the solid state; this is a form which is brought about when aggregate particles of extremely fine size, say less than 1 micron and particularly less than 0.5 micron, are dispersed in water; under these conditions, the aggregates form what are called gel particles, comprising particles so highly swollen that a question may arise as to whether they are in a solid or a liquid state. For the purposes of this invention, it is to be understood that the terms "aggregates", or "solid aggregates", or "solid particles", or "solid form", or "finely divided solid form" are intended to include these gel particles of aggregates as well as the aggregates of larger size.

WHAT WE CLAIM IS:—

1. A cosmetic composition containing cellulose crystallite aggregates as hereinbefore defined having an average level-off D.P. in the range of 15 to 375 anhydroglucose units and at least one active cosmetic ingredient compatible with the aggregates and present in the com-

- position in an available form and in an amount sufficient so as to impart its characteristic cosmetic effect or property when the composition is applied topically.
- 5 2. A cosmetic composition according to claim 1, wherein the cellulose crystallite aggregates are mechanically disintegrated.
- 10 3. A cosmetic composition according to claim 2, wherein at least 1% by weight, of the said cellulose crystallite aggregates have a particle size not exceeding 1 micron.
- 15 4. A cosmetic composition according to claim 1, 2 or 3, wherein the cellulose crystallite aggregates contain not over 500 ppm. of ether-extractable components.
5. A cosmetic composition according to any one of claims 1 to 4, and in an apparently dry powder form.
- 20 6. A cosmetic composition according to any one of claims 1 to 4, and in a compressed form.
- 25 7. A cosmetic composition according to any one of claims 1 to 4, wherein the composition includes water and is in a gel form, the cellulose crystallite aggregates being colloiddally dispersed in the water, and the gel comprising from 3% to 35%, by weight, of the aggregates.
- 30 8. A cosmetic composition according to any one of claims 1 to 4, wherein the composition includes water and is in a pourable liquid form, the cellulose crystallite aggregates being colloiddally dispersed in the water.
- 35 9. A cosmetic composition according to any one of claims 1 to 4, 7 and 8, and including an oleaginous material.
10. A cosmetic composition according to any one of claims 1 to 4, wherein the active cosmetic ingredient is an active detergent substance. 40
11. A cosmetic composition substantially as hereinbefore described with reference to Example 1. 40
12. A cosmetic composition substantially as hereinbefore described with reference to Example 2. 45
13. A cosmetic composition substantially as hereinbefore described with reference to Example 3. 50
14. A cosmetic composition substantially as hereinbefore described with reference to Example 4. 50
15. A cosmetic composition substantially as hereinbefore described with reference to Example 5. 55
16. A cosmetic composition substantially as hereinbefore described with reference to Example 6. 55
17. A method of making a cosmetic composition, substantially as hereinbefore described. 60

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